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ORIGINAL ARTICLE

A second life for mining waste as an amendment for soil remediation

Lorena Salgado[1,2] · Laura Aparicio[1] · Elías Afif[3] · Esther Fernández-López[1] · Jose R. Gallego[1] · Rubén Forján[1,3]

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Abstract

Mining is a cornerstone of the productive system, particularly in sectors, such as renewable energy and electronic equipment production, where its significance is anticipated to grow in the coming years. Aligned with the principles of Nature-Based Solutions and Zero Waste policies, recycling mining waste as soil amendments could concurrently restore degraded areas and reduce the disposal of mining waste. In this context, we aimed to remediate a post-mining soil primarily impacted by heavy metal pollution using an alkaline waste generated in dunite exploitation, either in combination with compost or independently in field conditions. The objectives were to minimize heavy metal(loid)s mobility (As, Cu, Cd, Ni, Pb, and Se), to improve soil health, and stimulate plant growth (phytoremediation, *Lolium perenne* L. was used). Results revealed that the combination of dunite and compost successfully reduced the concentrations of available Cu and Ni in the soil by more than four times, significantly enhanced soil properties, and promoted the harvest of a greater biomass. Additionally, *Lolium perenne* L. demonstrated phytostabilizing capacity for Cu and Ni in the soil treated with the amendments. In conclusion, the utilization of combined dunite-based and organic amendments proves to be a favorable strategy for restoring polluted post-mining soils.

Keywords Phytoremediation · Compost · Dunite · Mining soil · Nature-based solutions

Introduction

Mining plays a pivotal role in the societal development and is set to become increasingly critical in future, primarily due to the evolving dynamics of population growth and industrial expansion. Today, the global mining industry extracts more than 10 billion tons of raw materials [1] and is set to become a key activity in the necessary energy transition toward a more sustainable and environmentally friendly development [2, 3].

Contemporary trends in sustainable mining advocate for the concurrent restoration of tailings dumps, spoil heaps, and mining soils as the extractive operations progress, rather than waiting until mining closure [4]. This approach should be based on the application of cost-effective and sustainable technologies such as those included within the burgeoning concept of nature-based solutions (NBS) [5]. In this sense, the imperative to remediate mining soils may be easily linked to the European Commission's Zero Waste Policy [6], wherever the application of amendments derived from other mining or industrial waste is used.

Within the NBS, soil chemical stabilization [7] is one of the techniques that has gathered substantial attention in recent years. This approach may involve the design and application of soil amendments composed exclusively of various types of residues [8]. The primary goal of designing and implementing such amendments is to rectify the issues associated with degraded soil and subsequently establish a resilient vegetative cover [9, 10].

To reckon soil fully restored, it must be capable of independently sustaining a stable plant cover. In this context, it is common practice to select plant species with phytoremediation capabilities, capable of tolerating specific metal

✉ Rubén Forján
forjanruben@uniovi.es

[1] Environmental Biogeochemistry & Raw Materials Group and Institute of Natural Resources and Territorial Planning (INDUROT), University of Oviedo, Campus of Mieres, 33600 Mieres, Spain

[2] SMartForest Group, Department of Organisms and Systems Biology, Polytechnic School of Mieres, University of Oviedo, 33600 Mieres, Spain

[3] Plant Production Area, Department of Biology of Organisms and Systems Biology, University of Oviedo, 33600 Mieres, Spain

concentrations while assisting in their retention in the soil by reducing chemical mobility, i.e., species with phytostabilizing capacity [11] and not with phytoextracting potential. Furthermore, phytoremediation is a technique harmonious with the application of soil amendments and is encompassed within the framework of NBS. In this context, many studies have shown that the effectiveness of the joint application of chemical stabilization and phytoremediation (phytostabilization) can be successful [12–14]. For example, the application of different amendments combined with *Brassica juncea* L. is more effective than separate applications [15].

The reuse of mining waste within the same mining site or in other mines offers two distinct advantages. First, it reduces waste production, and second, it transforms this waste into new raw materials for the production of soil amendments [16, 17]. Some of these mining by-products may contribute valuable nutrients to the soil, enhancing cation exchange capacity and reducing the contents of (bio)available metals in the soil. Moreover, these inorganic by-products can be combined with organic amendments such as compost when necessary to obtain synergies [18, 19]. One particular mining by-product useful as an amendment in the remediation of contaminated soils is dunite waste. Dunite residues and by-products have shown potential as soil amendments due to their favorable composition, primarily consisting of basic cations, magnesium oxide, and lime. These components not only increase pH and limit metal mobility but also facilitate the establishment of plant cover as demonstrated in previous lab-scale studies [16, 20]. In this sense, a novelty of this study lies in its exploration of the real-world valorization of a mining by-product. Left unattended, such by-products would typically accumulate in vast landfills, posing potential environmental hazards in future.

Given the context provided in this introduction, the primary objective of this study is to develop dunite-based amendments, both independently and in conjunction with phytoremediation, to assess their impact on metal mobility, vegetation growth, and soil health in post-mining soils. The application of these treatments took place under field conditions as part of an in situ pilot-scale trial. In this way, we propose a remediation strategy that aligns seamlessly with the principles of NBS and Zero Waste guidelines.

Materials and methods

Soil

The post-mining soil (S) used in this study was selected from an area in northern Spain where open-pit mining of base and precious metals took place in the 1990s and 2000s. This soil presented (Table 1) high concentrations of some metals and metalloids, low levels of organic matter, and practical absence of vegetation.

Table 1 Characteristics and pollutant concentrations of polluted soil (S), compost (C), dunite (D), and biochar (B)

	Parameters	Units	S	C	D
	pH		7.89	7.28	9.23
	OM	%	1.04	44.27	0.52
	Ca^{2+}		16.46	228.34	1.21
	Mg^{2+}		7.76	35.1	10.1
	Na^{+}	$cmol_{(+)}kg^{-1}$	0.06	28.91	0.21
	K^{+}		0.94	27.41	0.41
	Al^{3+}		< DL	2.15	< DL
	CEC		24.86	322.6	11.93
Total	Ni	$mg.kg^{-1}$	27	29	1138
	Cu		505	201	29
	As		356	14	16
	Cd		39	1.68	0.9
	Pb		35	79	38
	Se		1	< DL	< DL
TCLP extraction	Ni		1.83	0.32	23.22
	Cu		21.19	2.78	< DL
	As		0.18	1.85	< DL
	Cd		0.09	< DL	< DL
	Pb		0.08	< DL	0.06
	Se		0.19	< DL	< DL

< *DL* below detection limit

Amendments

The main characteristics of the amendments, compost, and dunite used are shown in Table 1. This compost (C) is produced from sludge from wastewater treatment plants of the central area of Asturias (Spain) and shredded wood of mixed sources using the mechanically aerated piles technique. This compost was supplied by COGERSA (the company for regional waste management in Asturias). The dunite (D) utilized in this study corresponds to a residual fraction smaller than 63 microns, which currently holds no commercial value and is generated during the mineral processing procedure. This material was previously characterized in the studies carried out by [16] and [20].

Vegetation

The vegetation selected for this experiment was ryegrass (*Lolium perenne* L.). This species has phytoremediation capacity and is able to settle and spread easily in multiple environments [21, 22].

Experimental design and soil samples

A mixture of degraded soil and spoil from the old mining operation was excavated, homogenized, and distributed over a flat area in four plots of 2.5 m long, 1.5 m wide, and 0.5 m deep.

The first plot was kept as a control, while in the other three plots, 20% of the soil was replaced by amendments in the proportions shown in Table 2, with vigorous mixing to homogenize soil and amendments. The plots were then left undisturbed for stabilization for a period of 15 days until the ryegrass seeds were sown (sowing dose of 40 g m^{-2}). The experiment spanned a duration of 5 months, during which soil samples were collected at month 1 (T1) and at the final time point after 5 months (T2). Vegetation harvesting took place after the full 5-month period. Throughout the experiment, the plots remained under natural conditions without additional irrigation.

Composite samples were obtained from three sampling stations per plot. At each sampling station, a 1-kg composite sample, consisting of four increments from the top 5 cm of soil, was collected using a Dutch Edelman probe. The soil samples were air-dried at room temperature (< 25 °C), sieved through a 2-mm mesh, and material below 2 mm was quartered using a Jones riffle to obtain representative subsamples. The soil samples were conserved in sterilized plastic bags and stored at 4 °C until analyses.

Soil and amendment analysis

Concentration analyses for available and total levels of As, Cu, Cd, Ni, Pb, and Se were conducted on soil samples. Available concentrations were determined by means of TCLP extraction (Toxicity Characteristic Leaching Procedure) following the USEPA Method 1311 (1992), and the concentrations were determined by ICP-MS (Inductively Coupled Plasma-Mass Spectrometry), with detection limits of 0.02 mg kg^{-1} for As, Cu, Cd, Ni, Pb, and Se. Total concentrations were determined through an aliquot of 1 g soil (< 2 mm) that was ground in an agate mortar to achieve a particle size of < 100 µm. These solid samples were sent to an ISO 9002 and ISO-17025 accredited laboratory (Acmelabs, Vancouver) for chemical analysis and homogenized before performing the analyses. The total concentrations of As, Cu, Cd, Ni, Pb, and Se were determined by ICP-MS after aqua regia extraction, with detection limits of 2 mg kg^{-1} for As, 1 mg kg^{-1} for Cu, 0.5 mg kg^{-1} for Cd, 1 mg kg^{-1} for Ni, 2 mg kg^{-1} for Pb and 0.5 mg kg^{-1} for Se.

pH and electrical conductivity (EC) were determined using a multi-meter METTLER TOLEDO SevenCompact in a 1:2.5 water to soil extract (Porta, 1986). Exchangeable cations (Ca^{2+}, K^{+}, Mg^{2+}, Na^{+}, and Al^{3+}) were extracted with 0.1 M $BaCl_2$ [23] and concentrations were determined by ICP-MS. The effective cation exchange capacity (CEC) was calculated using the sum of exchangeable cation concentrations [24]. The organic matter content was obtained by Loss-On-Ignition Method (Lenton furnaces).

Table 2 Proportion of amendments and soil per plot

CODE	Soil (%)	Compost	Dunite	Vegetation
S	100			√
SD	80		20%	√
SC	80	20%		√
SCD	80	10%	10%	√

Factors for plant production

A series of critical values were assigned to each of the chemical parameters, based on the model of the SFCC (Soil Fertility Capability Classification) proposed [25], and adapted by [26]. These were used to evaluate the limiting factors for plant production:

- *K factor* indicates that a soil is K deficient if K^{+} < 0.2 $cmol_{(+)}kg.^{-1}$
- *Mg Factor* (Mg deficiency): this factor applies to soils where the level of available Mg^{2+} is not sufficient for the needs of the plants, which may be caused either by a low content of this element in the exchange complex (< 0,4 $cmol_{(+)}kg^{-1}$ or by an imbalance in the Ca^{2+}/Mg^{2+} ratio > 50).
- *Ca Factor* (Ca deficiency): when the value of the Ca^{2+} content in the exchange complex < 1.5 $cmol_{(+)}kg^{-1}$.
- *Na Factor*: The Na Factor is defined as the condition wherein Na saturation within the 50 cm surface exchange complex reaches or exceeds 15%.

Plant analysis

Once the vegetation was harvested, it was dried in an oven at 30 °C for a week. Subsequently, the entire sample was ground with a ZM 200 ultracentrifugal mill (Retsch, Verder Scientific GmbH & Co. KG, Haan), obtaining a homogeneous subsample whose grain size was below 40 µm. This subsample was subjected to acid digestion with a mixture of H_2O_2 and HNO_3 (1:6 v/v) in a microwave oven (Milestone ETHOS-1). Finally, the concentrations of metal(oid)s were determined by ICP-MS.

The Transfer Coefficient (TC) was calculated to determine the ability of ryegrass to accumulate or stabilize metal(oid)s (the plant is usually contemplated for phytoextraction when TC > 1) [27]. The TC is calculated in our case as the quotient between Ca (concentration of the element in

the aerial part of the plant) and the total concentration (Cs) of the element in the soil.

Statistical analysis

All analytical determinations were performed in triplicate. A statistical analysis was conducted to verify if there were statistically significant differences in the parameters analyzed between the soil and the various treatments across. The data obtained were statistically treated using the SPSS program version 24.0 for Windows. Analysis of variance (ANOVA) and test of homogeneity of variance were carried out. In the case of homogeneity, a post hoc least significant difference (LSD) test was performed. When there was no homogeneity, Dunnett's T3 test was performed. A correlated bivariate analysis was also carried out by means of Pearson correlation.

Results and discussion

Evolution of pH, exchange cations, and cation exchange capacity (CEC)

The control soil (S) and the soil in which the different treatments (SC, SD, SCD) were applied had a basic pH (between 7.27 and 8.36) throughout the experimental period (Table 3). However, at a statistical level, these soils showed significant differences in terms of pH values at time T2 when the control soil had the lowest pH values, lower also than at time T1 (Table 3). The SD treatment had the highest pH values (Table 3), probably due to the alkalinity of the dunite amendment (pH = 9.23 as shown in Table 1). In general terms, the pH range for plant production varies between 5.5 and 7.5 and soil pH greater than 8.0 is considered strongly alkaline for most crops [28]. The SD treatment was clearly above this range (pH = 8.36, Table 3).

At time T1 and T2, the control soil S had a lower CEC than the treatments (Table 3). At the end of the experimental time, the direct application of compost (SC) and dunite (SD) caused the highest increase in CEC (Table 3). In terms of CEC values, the control soil and the treatments exceeded the minimum values for vegetation establishment throughout the experimental time. It should be noted that these CEC values are due to the basic cations as the Al content remained below the detection limit. This increase in CEC due to basic cations may be one of the reasons for the increase in pH values in the treatments (SC, SD, SCD) as the behavior of pH is usually related to variations in CEC [29].

Regarding factors for plant production, the soil S is affected by the K factor due to its low K content. Despite the high Ca cation content, neither the control soil or the treatments would be affected by the Mg factor or the Ca factor. Finally, both the control soil and the treatments would be Na deficient as their Na content is below 15% in relation to the CEC.

Evolution of organic matter content

As anticipated, both the control soil (S) and the direct addition of dunite (SD) exhibited the lowest OM content compared to the addition of compost (SC) or compost mixed with dunite (SCD) (Fig. 1). The control soil was characterized as a clayey inorganic soil [30], and the by-product derived from dunite is also a purely inorganic amendment [16]; therefore, as indicated in Table 1, these materials

Table 3 Evolution of pH, exchange cations and cation exchange capacity (CEC)

			S	SC	SD	SDC
Time 1	pH		7.75 ± 0.17a	7.95 ± 0.10a	7.79 ± 0.15a	8.07 ± 0.27a
	Na^+	$cmol_{(+)}kg^{-1}$	< DL	0.07 ± 0.01c	4.90 ± 0.16a	3.57 ± 0.12b
	Mg^{2+}		8.15 ± 0.09b	6.98 ± 0.55b	14.07 ± 0.46a	12.08 ± 0.60a
	K^+		0.26 ± 0.01d	0.66 ± 0.02c	6.87 ± 0.25a	5.44 ± 0.08b
	Ca^{2+}		7.05 ± 0.01d	18.74 ± .88c	109.11 ± 1.98a	75.52 ± 3.23b
	Al^{3+}		< DL	< DL	< DL	< DL
	CEC		15.46 ± 0.07d	26.46 ± 1.46c	134.97 ± 2.86a	96.62 ± 4.04b
Time 2	pH		7.27 ± 0.08c	7.44 ± 0.18bc	8.36 ± 0.11a	7.71 ± 0.04b
	Na^+	$cmol_{(+)}kg^{-1}$	< DL	1.44 ± 0.03a	0.44 ± 0.03b	2.89 ± 0.09c
	Mg^{2+}		8.21 ± 0.23b	10.28 ± 0.30a	7.76 ± 0.48b	2.14 ± 0.26
	K^+		0.65 ± 0.04c	4.11 ± 0.13a	2.15 ± 0.15b	0.23 ± 0.02d
	Ca^{2+}		18.54 ± 0.51c	107.03 ± 2.45a	97.40 ± 6.21a	36.01 ± 0.60b
	Al^{3+}		< DL	< DL	< DL	< DL
	CEC		27.42 ± 0.70c	122.88 ± 2.39a	107.77 ± 6.88a	75.39 ± 0.79b

For each row, different letters in different samples mean significant differences (n = 3, ANOVA; $p < 0.05$). < DL: below detection limit. Standard deviation is represented by ±

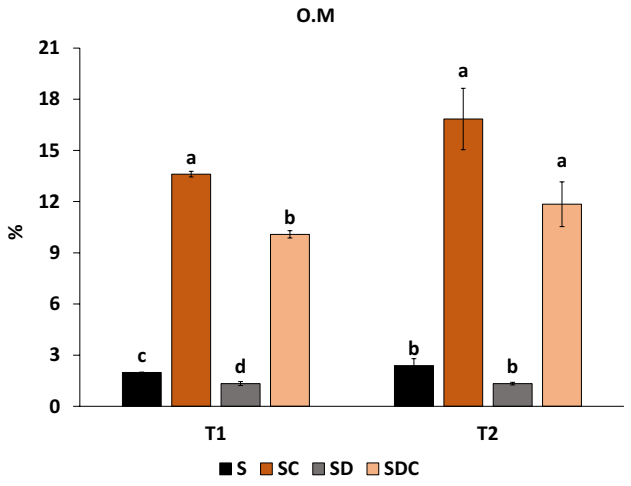

Fig. 1 Evolution of soil organic matter content. Different letters in the bars (for each sampling time) indicate significantly different results ($P < 0.05$)

initially had very low levels of OM. Evidently, the increase in OM observed in SC and SCD treatments was attributable to the addition of compost (Fig. 1).

In the same context, it is noteworthy that, by the end of the experimental period, no significant differences in OM were observed between the SC and SDC treatments. This may be attributed to the fact that, although the SDC treatment was formulated with less compost than the SC treatment, the organic matter in the compost could have been stabilized by forming organo-mineral complexes with the dunite [31]. The increase of OM in the soil is crucial, as directly influences various soil factors, such as humic and fulvic acid content [32], water holding capacity [33], soil structure [34], soil microbial community [35], nutrient supply [36], and usually decreases the availability of potentially toxic elements [37], all of which directly or indirectly influence plant production [38].

Evolution of pollutants extractable TCLP concentrations

As shown in Table 1, in the study soil (S), the available concentrations (extractable TCLP) of elements, such as As (0.18 $mg.kg^{-1}$), Cd (0.09 $mg.kg^{-1}$), Pb (0.08 $mg.kg^{-1}$), or Se (0.19 $mg.kg^{-1}$), were very low, which is not the case for Cu (21.19 $mg.kg^{-1}$). Also, in the dunite residue, there is a notable presence of Ni, both total and available. Consequently, the performance of the amendments, in terms of reduction of metal(loid)s availability, has solely been focused on Cu and Ni.

As referred above, the element with the highest available concentrations was Cu. As shown in Fig. 2A, the available concentrations of Cu increased between time T1 and T2, which indicates that this type of soil should be treated to prevent an increase in the mobility. This higher Cu availability in the S soil is possibly due to the lack of OM and a low CEC. In this sense, significantly negative correlations were found between available Cu concentrations and OM content (− 0.74, $P < 0.01$) and CEC (− 0.78, $P < 0.01$). The amendments reduced the available Cu concentrations throughout the experimental time; specifically, at time T2, it can be observed how compost (with or without dunite) was more effective. This better yield may be related to the high levels of OM in the compost as OM has a remarkable Cu sorption capacity and therefore reduces its availability [39, 40]. Also, different authors have concluded that the increase of CEC by basic cations (as happens when dunite is used) favor the retention of metal cations in the soil [41, 42].

In turn, the dunite residue had considerable total and available Ni concentrations as abovementioned (Table 1), and the origin of Ni in the dunite residues is related with the mineralogy of the rock [16]. It should be noted that when the dunite residues are mixed with compost, the available Ni concentrations decrease significantly; note that the concentrations in the SDC treatment are statistically similar to those in the control soil (Fig. 2B). At the end of the experimental time, the SD treatment had a higher CEC than the SDC treatment (Table 3), which strongly suggests that the decrease in available Ni concentrations is due to the increase of OM caused by the addition of compost (Fig. 2B). On the whole, the available Ni concentrations in the control soil and the amendments were significantly negatively correlated with the OM content (-0.65, $P < 0.01$). Authors such as [43, 44] concluded that decreases in available Ni concentrations are mainly due to the increase in pH, OM and CEC; in our case, the most differential factor was the contribution of OM. This positive effect may be attributed to the fact that composted residues have a strong affinity for the sequestration of heavy metals such as Ni. Compost enhances the complexation of heavy metals in organic molecules, thereby limiting their solubility and potential bioavailability in the soil [45, 46].

Metals in plant tissues

The addition of compost (with or without dunite) resulted in a clear increase in fresh biomass and height of ryegrass (Table 4). The lowest biomass was harvested in the SD treatment (Table 4), which indicates that this type of soil needs not only nutrients but also OM for proper plant growth. In this work, the increase in harvested biomass is significantly positively correlated with the increase in CEC (0.62, $P < 0.05$) and OM content (0.87; $P < 0.01$) as previously reported [10]. It is also evident that the nutrient supply from the dunite is clearly improved by combining dunite with compost [20].

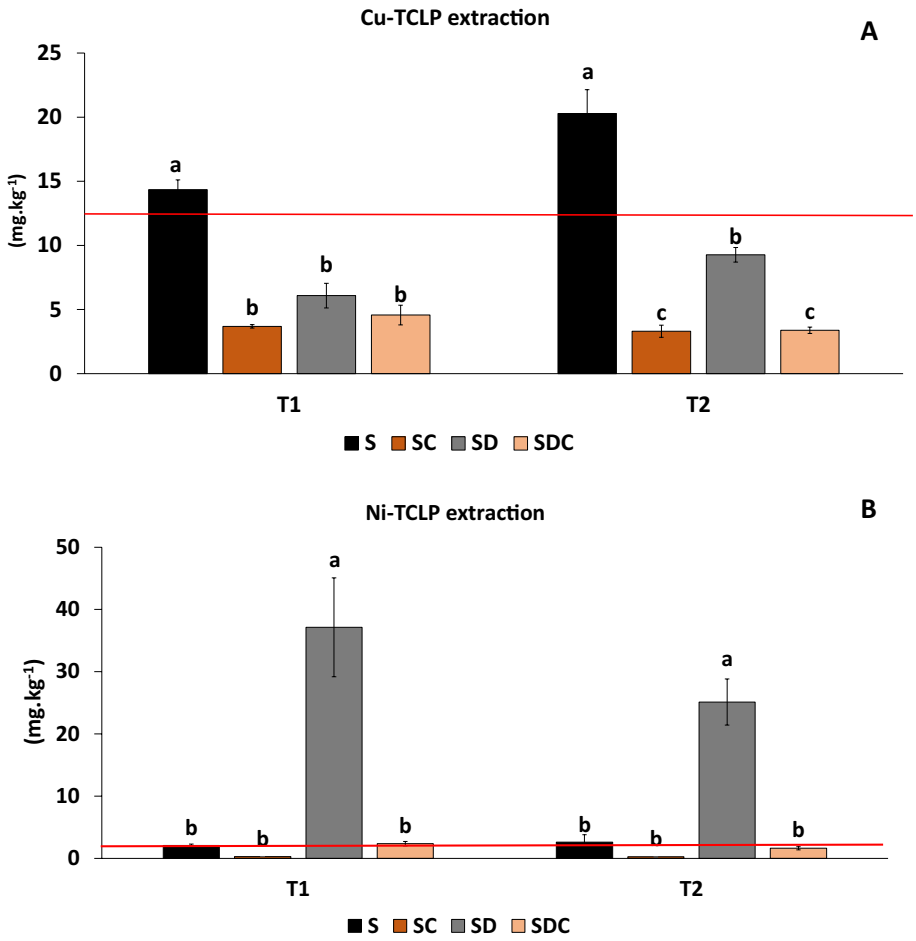

Fig. 2 Evolution of (available) TCLP-extractable concentrations of Cu (**A**) and Ni (**B**) in the control soil and the treatments. Different letters in the bars (for each sampling time) indicate significantly different results ($P < 0.05$). The red line represents the values of available Cu and Ni (TCLP extracted) in the original soil

Table 4 Harvestable amounts of Cu and Ni and determination of metals in plant tissues

		S	SC	SD	SDC
Dry biomass	g	70.06 ± 0.04c	861.08 ± 0.13a	62.41 ± 0.52d	211.00 ± 0.02b
height	cm	3.11 ± 0.09c	27.20 ± 0.19a	3.12 ± 0.10c	21.18 ± 0.05b
Cu	$mg.kg^{-1}$	45.72 ± 0.15c	58.67 ± 0.08a	52.34 ± 0.29b	52.55 ± 0.12b
Ni		27.96 ± 0.08d	33.14 ± 0.65c	42.66 ± 0.31b	44.36 ± 0.19a
Cu	**TC**	0.17 ± 0.01b	0.12 ± 0.01c	0.24 ± 0.00a	0.11 ± 0.00c
Ni		1.36 ± 0.20a	0.99 ± 0.07a	0.05 ± 0.01b	0.11 ± 0.02b

u.l. undetectable level

For each row, different letters in different samples mean significant differences ($n = 3$, ANOVA; $p < 0.05$)

Harvestable Cu and Ni contents were higher in the amended soils than in the S control soil (Table 3). This may be because the amendments improved soil conditions for plant growth, especially in the case of SC and SDC, so the plants grow healthier and could tolerate more Cu and Ni content. However, in the results corresponding to the TC factor, it was observed that these contents were not transferred to the aerial part but remained in the roots (Table 4). This suggests that ryegrass adopted a phytostabilizing strategy in this experiments [27], and thus Cu is not transferred to the trophic chain. In this sense, phytostabilization is an appropriate technology for large, degraded areas [47–49]. Conversely, for Ni, the ryegrass harvested on the control soil revealed phytoextracting characteristics as $TC > 1$ [27]. However, any amendment addition promoted a reduction of TC below 1 (Table 4).

Conclusions

The application of dunite combined with compost under pilot scale and field conditions has yielded promising results, providing insights into its potential impact on soil health

and vegetation growth. In terms of reducing available Cu and Ni concentrations, the dunite and compost amending demonstrated the highest effectiveness. This effect was not attributed to pH variation, as both the soil and the amendments used presented alkaline pH, i.e., the reduction in Cu and Ni availability was attributed to the increase of CEC and OM content. On the contrary, the application of dunite alone proved unfavorable, resulting in increased available concentrations of Ni.

The enhanced soil conditions following the application of the dunite-compost amendment also contributed to healthier plant growth and increased biomass. Furthermore, under these settings, vegetation primarily exhibited phytostabilizing characteristics for Cu and Ni, a critical factor in preventing metals from entering the food chain, whereas plants harvested from the control soil (S) transferred Ni to the aerial parts, posing associated risks that can be easily limited by amendments addition (compost, dunite, or the combination). To extrapolate this study and similar amendments to other soil types, it is necessary to consider the physicochemical characteristics of specific soils to ensure comparable results. Insights derived from this research provide valuable guidance for promoting sustainable mining practices, aligning with the principles of nature-based solutions (NBS) and zero waste regulations. By employing innovative approaches such as those proposed here, there exists an opportunity to foster more resilient and environmentally responsible mining industry.

Author contributions Lorena Salgado — conceptualization, study design, data acquisition, data analysis, lead writing, editing, supervision. Laura Aparicio — data acquisition, data analysis, lead writing. Elías Afif — conceptualization, data analysis, writing, review. Esther Fernández-López — data acquisition, writing, review. Jose R.Gallego — conceptualization, editing, review, founding acquisition. Rubén Forján — conceptualization, study design, data acquisition, writing, editing, review.

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Data availability The datasets generated and analyzed during the current study are available from the corresponding author on reasonable request.

Declarations

Conflict of interest The authors declare no competing interests.

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